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Bond Strength of Resin Cements to Dentin Using New Universal Bonding Agents

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Bond Strength of Resin Cements to Dentin Using New Universal Bonding Agents

ABSTRACT

The purpose of this study was to determine the effect of new universal bonding agents on the bond strength of dual-cure resin cements to dentin. One hundred forty extracted human third molars were mounted in dental stone and sectioned with a saw to remove coronal tooth structure. The teeth were randomly divided into seven groups of twenty based on the use of five universal bonding agents (All-Bond Universal, Bisco; FuturaBond U, VOCO; Prime&Bond Elect, Dentsply; Scotchbond Universal, 3M ESPE; Clearfil Universal, Kuraray) compared to two self-etch bonding agents (Clearfil SE Bond and Clearfil SE Bond 2, Kuraray). Each group was further divided into two equal subgroups of ten specimens each with each subgroup tested with either self- or light-cure activation of the dual-cure resin cement (Calibra, Dentsply). The bonding agent was applied per manufacturers' instructions to the dentin surface of each specimen. The specimens were placed into a jig and resin cement was inserted into the mold to a height of 3-4 mm and light cured. Specimens were stored for 24 hours in 37^{0}C distilled water and tested in shear in a universal testing machine. A mean shear bond strength value (MPa) and standard deviation was determined per group. Data were analyzed with 2-way ANOVA/Tukey's (alpha=0.05). A significant difference was found between groups based on bonding agent ($p<0.0001$) or curing mode ($p=0.026$) with no significant interaction ($p=0.117$). Light curing of the resin cement in dual-cure mode resulted in significantly greater bond strengths than if the cement was allowed to self-cure. The new simplified universal bonding agents resulted in significantly lower shear bond strength of the resin cement to dentin than the non-simplified, two-step, self-etching bonding agents Clearfil SE Bond or Clearfil SE Bond 2.

INTRODUCTION

Advances in dental material technology ultimately strive to improve the care rendered to patients by providing services that are increasingly effective. Materials continue to evolve, generally yielding more esthetic, stronger, longer-lasting, and simpler to use products. Adhesive technology specifically is an area aggressively pursued by manufacturers who recognize that dentists desire to have a simple bonding agent that can be used for all indications.

The goal of dental adhesives is to provide an equally effective bond to two hard tissues of different nature (Van Meerbeek, 2011). Bonding to enamel has been proven to be durable but bonding to dentin has been shown to be far more intricate and can apparently only be achieved

when more complicated and time-consuming application procedures are followed (Van Meerbeek, et al., 2011).

Today's adhesives either follow an 'etch-and-rinse' or a 'self-etch' approach and both techniques have been shown to perform successfully in laboratory and clinical settings (Van Meerbeek, et al., 2011). The initial etch-and-rinse adhesives required three steps that included an acidic conditioner, primer and adhesive monomer. Three-step, etch-and-rinse adhesives (e.g., Optioned FL, Kerr, Orange, CA) have been proven to be versatile and clinically successful De Munck et al., 2005, Peumans et al., 2014). The trend, however, has been to develop systems that are "simplified", or in other words, involve fewer steps with less procedure time (Tay et al., 2002). The manufacturers combined the primer and the resin monomer components of the three-step etch-and-rinse adhesives to reduce the number of steps from three to two.

With self-etch adhesives, the acidic conditioner and primer are combined into one self-etching primer component. Self-etch adhesives are available as two-step and one-step adhesives, depending on whether the self-etching primer and adhesive resin are provided separately or are combined into one single solution (Van Meerbeek, et al., 2011). The simplified one-step systems are a mix of hydrophilic and hydrophobic components and have been documented to have several shortcomings to include a reduced 'immediate' bond strength and lower bond strength over time compared to multi-step, non-simplified adhesives. An example of a clinically successful non-simplified, self-etch adhesive is Clearfil SE Bond (Kuraray, New York, NY) (Van Meerbeek, et al., 2011). In a recent systematic review of the literature, Peumans et al. (2014) reported Clearfil SE Bond to be the most effective clinically with an annual failure rate of only 2.5% when restoring non-carious cervical lesions.

In addition to lower bond strengths, simplified adhesive systems (i.e., two-step etch-and-rinse, one-step self-etch) have been shown to have potential incompatibilities with self-curing composite materials. Low bond strength between self-curing composite resins, such as resin cements and core materials, and acidic simplified adhesives is a well-studied phenomenon (Kanehira et al., 2006). A chemical incompatibility may occur in the oxygen-inhibited surface layer when the acidic monomers of the simplified adhesives interact in an acid-base reaction with the aromatic tertiary amine of the self-cure composite. The amine activator is no longer able to initiate the chemically activated free-radical polymerization, resulting in incomplete polymerization and compromised bond strengths along the resin-adhesive interface (Bowen et al., 1982; Ikemura et al., 1999; Nakamura et al., 1985). It has also been found that if a dual-cure composite material is not sufficiently light cured, this incompatibility may also occur (Tay et al., 2003). Another reason for incompatibility stems from the hydrophilic mix created by

incorporating hydrophilic monomers in the simplified primer-adhesive combination. Permeability of water from hydrated dentin migrates across the adhesive interface and accumulates as fine water blisters (Kanehira et al., 2006). The permeated water is described as water trees under transmission electron microscopy and may contribute to diminished bond strengths of self-cure composite materials or dual-cure materials that are not adequately light cured (Tay et al., 2003).

Most resin cement systems that use self-cure modalities indicate their use exclusively with non-simplified etch-and-rinse or self-etch adhesives. Manufacturers typically recommended adequate light curing or the use of an additional dual-cure activator if unable to do so. However, studies have shown that the use of activators does not completely eliminate this incompatibility (Tay et al., 2003).

Recently, new “universal adhesives” have been introduced. These universal adhesives are simplified bonding agents that are indicated to be used with either self-etch, etch-and-rinse, or selective enamel etching techniques (Perdigao and Loguercio, 2014). All current universal adhesives are simplified adhesives that must contain water, which is required for the ionization of the hydrophilic acidic monomers when used in the self-etch mode. The hydrophilicity of simplified adhesives makes them behave as semi-permeable membranes, allowing fluid transudation across the resin/dentin interface, which leads to bond degradation (Perdigao and Loguercio, 2014). A recent laboratory study has shown that universal adhesives demonstrate signs of degradation after 12-months of water storage when applied either in a self-etch or etch-and-rinse mode (Marchesi, et al., 2013). A definition for the term “universal adhesive” was proposed by Dr. Byong In Suh (the CEO of Bisco who manufactures ALL-BOND Universal) and is as follows: 1) hydrophobic enough to be used as a single-layer adhesive, 2) can be used with any etching technique: etch-and-rinse, self-etch, or selective-etch modes, 3) indicated for both direct and indirect clinical procedures using light-cure, dual-cure, or self-cure materials (preferably without the need for a separate dual-cure activator), 4) can bond to indirect substrates (except for silica-based ceramics) because of low film thickness (<10um) (Suh, 2013).

A common material that is bonded with adhesives is dual-curing resin cements. To date no studies were found in the literature that have evaluated the shear bond strengths of self-activated dual-cure resin cements to dentin using new universal adhesive bonding agents that are currently available on the market. Recently, Kuraray has introduced Clearfil SE Bond 2, a two-step, self-etch, light-cure bonding agent with a new catalyst system and dual-cure activator for use with indirect restorations and core build-up materials (Kuraray website). No studies have been published evaluating the bond strength of this new non-simplified adhesive to dentin

using resin cements. It would be valuable to compare the new simplified universal bonding agents to the non-simplified adhesive bonding agents so that their potential efficacy in clinical practice can be better understood.

Two null hypotheses were tested. First, that there would be no difference in the shear bond strength of a dual-cure resin cement to dentin based on type of adhesive bonding agent. Second, that there would be no difference in the shear bond strength of a dual-cure resin cement to dentin based on type of curing mode.

MATERIALS AND METHODS

The protocol used for this study was approved by Institutional Review Board at Wilford Hall Ambulatory Surgical Center, JBSA-Lackland, Texas. One hundred and forty extracted human third molars stored in 0.5% Chloramine-T at 4 degrees C were used within 6 months following extraction. Teeth with carious lesions were excluded. The teeth were mounted in dental stone in PVC pipes with the crown exposed and accessible. A diamond saw (Isomet, Buehler, Lake Forest, IL) was used to remove 2mm or more coronal tooth structure to ensure dentin exposure and the proper orientation of the surface relative to the direction of the applied shear force. Each specimen was examined under a stereomicroscope (SMZ-1B, Nikon, Melville, NY) at 10X magnification to ensure complete exposure of the dentin surface with no residual enamel. A uniform smear layer was created on the flat dentin surfaces using ten passes on 600-grit silicon carbide paper.

The 140 mounted specimens were randomly divided into seven groups (20 specimens to each group) based on the use of seven bonding agents: ALL-BOND Universal (Bisco, Schaumburg, IL), Futurabond U (VOCO, Briarcliff Manor, NY), Prime&Bond Elect (Dentsply, York, PA), Scotchbond Universal (3M ESPE, St. Paul, MN), Clearfil Universal (Kuraray), Clearfil SE Bond (Kuraray), and Clearfil SE Bond 2 (Kuraray). All bonding agents with the exception of Clearfil SE Bond and Clearfil SE Bond 2, both two-step self-etch bonding agents, are marketed as universal bonding agents. Notably, Clearfil SE Bond is not indicated per the manufacturer's instructions for use with indirect clinical procedures and self-cure resin cements. Clearfil SE Bond 2, however, is indicated for use with indirect clinical procedures and self-cure resin cements, and it includes a dual-cure activator to be used in this application.

The bonding agents were applied to the dentinal surface according to manufacturer's instructions (see Table 1). The adhesives were cured as recommended by the manufacturer using the Bluephase G2 (Ivoclar Vivadent, Amherst, NY) light-curing unit. Irradiance of the

curing light was monitored with a radiometer (LED Radiometer, Kerr) to verify irradiance levels above 1200 mW/cm^2. Each of the seven groups was further divided into 2 equal subgroups of ten specimens, with each subgroup tested with either self- or light-cure activation mode. The resin cement chosen for this study was Calibra (Dentsply). Calibra is a resin cement which can be used in light-, self- or dual-cure modes. The manufacturer contraindicates the use of Calibra in pure self-cure applications (limited or no light curing) with simplified self-etch or etch-and-rinse bonding agents in the absence of a dual-cure activator (Reference Dentsply Website Instructions). The 140 specimens were randomly assigned to 14 treatment subgroups of 10 specimens each. The specimens were placed in an Ultradent Jig and secured beneath the white non-stick Delrin insert (Ultradent, South Jordan, UT). The resin cement was mixed and applied into the mold according to manufacturer's instructions to a height of 3-4mm. The bonding area was limited to a 2.4mm diameter circle determined by the Delrin insert. The self-cure subgroups were allowed to self-cure undisturbed for a period of 15 minutes in a light-proof container. The light-cure subgroups were incrementally placed in 2mm increments with each increment cured for 20 seconds, as per manufacturer's instructions. Following the application of the resin cement with the designated curing method, all specimens were stored for 24 hours in distilled water at 37 degrees centigrade. The specimens were then loaded perpendicularly with a customized probe (Ultradent) in a universal testing machine (Instron Model 5943, Norwood, MA) using a crosshead speed of 1.0 mm/min until bonding failure occurred. Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure (Newtons) divided by the specimen surface area. The mean and standard deviation were determined for each group. Following testing, each specimen was examined using 10X stereomicroscope to determine failure mode as either: 1) adhesive fracture at the cement/adhesive/dentin interface, 2) cohesive fracture in cement, 3) mixed (combined adhesive and cohesive) in the cement and bonded interface or dentin and bonded interface, or 4) cohesive fracture in dentin.

Data were analyzed with a 2-way ANOVA and Tukey's post test to examine the effects of bonding agents (5-levels) or curing mode (2-levels) on the shear bond strength of resin cement to dentin (alpha = 0.05).

RESULTS

A two-way ANOVA statistical analysis was completed and a significant difference in shear bond strength was found between groups based on bonding agent ($p<0.0001$) or curing mode ($p=0.026$) with no significant interaction ($p=0.117$).

The new simplified universal bonding agents resulted in significantly lower shear bond strength of the resin cement to dentin than the non-simplified, two-step, self-etching bonding agents, Clearfil SE Bond or Clearfil SE Bond 2 (see Table 2). Light curing of the resin cement in dual-cure mode resulted in significantly greater bond strengths than if the cement was allowed to self-cure. Prime&Bond Elect had the lowest shear bond strength to dentin but it was not significantly different from FuturaBond U or All-Bond Universal. Clearfil Universal performed more moderately but was not significantly different from Scotchbond Universal. Clearfil SE Bond 2 had the highest bond strength to dentin and was significantly greater than Clearfil SE Bond, which was significantly greater than all of the other universal bonding agents. The universal bonding agents were associated with more adhesive failures compared to the non-simplified bonding agents, Clearfil SE Bond or Clearfil SE Bond 2 (see Figure 1).

DISCUSSION

The first null hypothesis was rejected. The results of this study indicate that the new universal adhesives have significantly lower shear bond strengths to dentin than non-simplified, two-step, self-etching bonding agents Clearfil SE or Clearfil SE 2. The second null hypothesis was also rejected. Light curing of the resin cement in dual-cure mode resulted in significantly greater bond strengths than if the cement was allowed to self-cure.

The universal adhesives tested in this study had more adhesive failures than the non-simplified adhesives tested, which suggests a weaker bonding interface between the resin cement and dentin (see Figure 1) (Al-Salehi and Burke, 1997). Notably, all specimens using Clearfil SE 2 failed in mixed modes and had no adhesive failures.

This study did not test the variable of whether a dual-cure activator was used or not. Therefore, within the limitations of this study no statements about the efficacy of dual-cure activators can be made. All adhesives were used as indicated by the manufacturers' instructions and therefore a dual-cure activator was used when indicated.

No published research has examined the bond strengths of resin cements to dentin using universal adhesives. A recent study by Munoz et al. evaluated the microtensile bond strength, nanoleakage, and degree of conversion within the hybrid layer for both etch-and rinse and self-etch strategies of universal simplified adhesive systems when used to bond a microhybrid composite restoration to dentin (Munoz et al., 2013). Only one of the universal adhesives

evaluated, Peak Universal Adhesive System, in both the etch-and-rinse and self-etch techniques, showed mean microtensile bond strengths statistically similar to the control, Clearfil SE Bond. However, it must be noted that of the three universal adhesives that they tested, only Peak Universal has a separate primer step and the other two universal adhesives are one-bottle systems with combined primer and adhesive.

The goals of universal adhesives are broad reaching. They have been described to be used for both direct and indirect restorations, compatible with any curing mode for resin-based materials, have the ability to bond to any substrate, and packaged in a single-bottle, no-mix, adhesive system that can be used in etch-and-rinse, self-etch, or selective-etch mode depending on the specific clinical situation and personal preference of the operator (Alex 2015, Suh 2013). They must also have some hydrophilic character in order to wet dentin, yet at the same time be as hydrophobic as possible to discourage hydrolysis and water sorption over time. The film thickness of the polymerized adhesive must not interfere with seating of indirect restorations. Also, universal adhesives must be acidic enough to be effective in a self-etching mode but not so acidic that they breakdown initiators needed for the polymerization of self- and dual-cure cements (Alex 2015). They must also contain water to allow the dissociation of the acidic functional monomers that makes self-etching possible (Alex 2015). The required use of water contributes to phase separation of the monomers, decreased shelf-life, and makes it difficult to complete the air-drying step before polymerization which could result in incomplete adhesive polymerization. (Alex 2015; Nishiyama et al., 2006; Moszner et al., 2005).

The unofficial definition of universal adhesives conflicts with some of the available products that are marketed as universal adhesives today. For example, the manufacturers of some universal adhesives recommend the use of separate primers to optimize bond strength to substrates other than dentin or enamel. Additionally, many universal adhesives are not one-bottle systems and require a separate dual-cure activator that must be mixed with the adhesive immediately before application.

Monomers with phosphate esters are the primary adhesive functional monomers used in current universal adhesive systems. 10-MDP (methacryloyloxy-decyl-dihydrogen phosphate) is a versatile amphiphilic functional monomer and is the most commonly used monomer used in current universal adhesives (Alex 2015). Of all functional monomers typically used in adhesives, 10-MDP is the most hydrophobic which may be important in terms of durability against water sorption and hydrolytic breakdown of the adhesive interface over time (Suh 2013; DeMunck et al., 2005; Hashimoto et al., 2004). Kuraray developed 10-MDP in the early 1980’s and their patent expired in 2003. Since then other manufacturers have created many products

using it. 10-MDP is structured with a hydrophobic methacrylate group on one end capable of bonding to methacrylate-based restorative materials and a hydrophilic polar phosphate group on the other end capable of chemical bonding to tooth tissues, metal, and zirconia (Alex 2015). 10-MDP is also capable of forming layers of stable MDP-calcium salts via ionic bonding to calcium in hydroxyapatite (Alex 2015). Alternative phosphate esters to 10-MDP such as PENTA-P (dipentaerythritol pent acrylate monphosphate) and GPDM (glycero-phosphate dimethacrylate) have also shown promise (Alex 2015, Suh 2011). The stability of the bond between resin-dentin and degree of nanoleakage of universal adhesives has recently been evaluated and it was found that MDP-containing universal adhesives showed bond strength stability at 6 months of water storage similar to a non-simplified self-etch adhesive, Clearfil SE Bond (Munoz et al., 2014). Studies have demonstrated that 10-MDP allows for a stable chemical bond to dentin over time both *in vitro* and *in vivo* due to the formation of a stable nano-hybrid layer together with a deposition of stable MDP-Ca salts at the adhesive interface (Munoz et al., 2013). All the bonding agents evaluated in this study contain 10-MDP except for FuturaBond U and Prime&Bond Elect. FuturaBond U contains a “modified” 10-MDP and Prime&Bond Elect contains Penta-P as their functional phosphate-ester group. FuturaBond U and Prime&Bond Elect had the lowest mean shear bond strength values of the bonding agents tested.

Simplified adhesive systems, which include universal adhesives, have been shown to have potential incompatibilities with self-curing resin materials. Low bond strength between self-curing resin materials and acidic simplified adhesives is a well-studied phenomenon (Kanehira et al., 2006). Two significant incompatibilities exist with the use of simplified adhesives with resin materials in self-cure mode, resulting in significantly lower bond strengths compared to specimens that are light- or dual-cured (Kanehira et al., 2006; Shade et al., 2014; Tay et al., 2003 Part 1 and Part 2; VanMeerbeek et al., 2011) and clinicians should be urged to consider the use of non-simplified bonding agents, especially in cases where complete light curing is not possible.

The first significant incompatibility with simplified systems is that the polymerized adhesive is an inherently hydrophilic and relatively hypertonic medium and may trigger osmotic fluid transport like a semipermeable membrane, enabling the transudation of water from the underlying dentin across an osmotic gradient toward the oxygen-inhibited adhesive agent–resin interface (Shade et al., 2014; Eick et al., 1997). The water diffusion from dentin accumulates in fine water blisters and leads to hydrolysis and reduced bond strength at the restorative interface (Alex 2015; Kanehira et al.; 2006, Tay et al., 2003). Manufacturers of universal adhesive systems attempt to address this problem by blending together monomers such as bis-GMA

(hydrophobic) and HEMA (hydrophilic) and other proprietary functional monomers (Alex 2015; Suh 2011).

The second significant incompatibility occurs in the oxygen-inhibited surface layer when the acidic monomers of the simplified adhesives interact in an acid-base reaction with the aromatic tertiary amine activator of the self-cure resin material. The acid-base reaction deactivates the aromatic tertiary amines which subsequently blocks initiation of chemically activated free-radical polymerization, resulting in incomplete polymerization and compromised bond strengths along the resin-adhesive interface (Bowen et al., 1982; Ikemura et al., 1999; Nakamura et al., 1985; Shade et al., 2014; Suh 2003). There is evidence that there is a direct correlation between pH and the compatibility with self- and dual-cure resin cements and composites and, in general, the more acidic the adhesive then the less compatible it is with self- or dual-cure resin materials (Suh 2003; Schittly et al., 2010). The deactivation of the amine activators in self-cured composites is considered to be the major cause of bond strength reduction, whereas permeability of the adhesives to water causes only a minor reduction in bond strength (Shade et al., 2014; Tay et al., 2003 Part 1 and Part 2).

To attempt to overcome the incompatibility of simplified adhesives with self-cure resin-based cements many manufacturers require separate “dual-cure activators”, usually arylsulfinate salts. Arylsulfinate salts are added so that they can be used as electron donors in initiator systems for free radical polymerization reactions (US Patent 2009). Some cements are amine-free and do not require use of separate “dual-cure activators.” All-Bond Universal and Futurabond U are unique in that the manufacturer does not require the use of separate “dual-cure activators” when used to self- or dual-cure resin-base materials. All-Bond Universal reportedly does not require a “dual-cure activator” because it is much less acidic than other universal adhesives at a pH of 3.2 (Alex 2015). Futurabond U reportedly does not require a separate “dual-cure activator” because it is already incorporated into the adhesive itself (Alex 2015).

In this study, Clearfil SE Bond 2 was found to have the highest shear bond strength to dentin when used with Calibra resin cement. Additionally, it was the only adhesive tested that had only mixed failures and no purely adhesive failures regardless of the self- or dual-cure mode tested. Clearly SE Bond 2 is marketed as a “truly universal adhesive” by Kuraray because it is able to be used with self- and dual-cure resin materials (when used with a proprietary dual-cure activator) as opposed to Clearfil SE Bond. According to Kuraray, the difference between Clearfil SE Bond and Clearfil SE Bond 2 is a new integrated photo-initiator chemistry which provides more free radicals when curing, which relates to higher monomer

conversion rates and stronger bonds (Kuraray website). Within the limitations of this in-vitro study, Clearfil SE Bond 2 was shown to perform superiorly to a gold-standard adhesive (Clearfil SE Bond) when bonding a resin cement to dentin. Ultimately, clinical trials will need to be completed to evaluate the effectiveness of Clearfil SE Bond 2.

Adhesive dentistry is complicated by difficult chemistry, varying types of substrates, and the need to follow precise clinical protocols. It is clear that the goal of current universal adhesives is to create a simple, easy to use bonding agent that can be used for all indications with any substrate. With very limited clinical trials evaluating the effectiveness of universal bonding agents, providers should remain skeptical that they will outperform more traditional non-simplified adhesive systems.

CONCLUSIONS

Within the limitations of this study, the following statements can be made. Dentin bond strengths can be maximized when the dual-cure resin cement is light-cured compared to relying on self-curing mechanisms alone. Significantly lower bond strengths to dentin were observed when universal adhesives were used with a resin cement in both dual-cure and self-cure mode compared to non-simplified adhesives.

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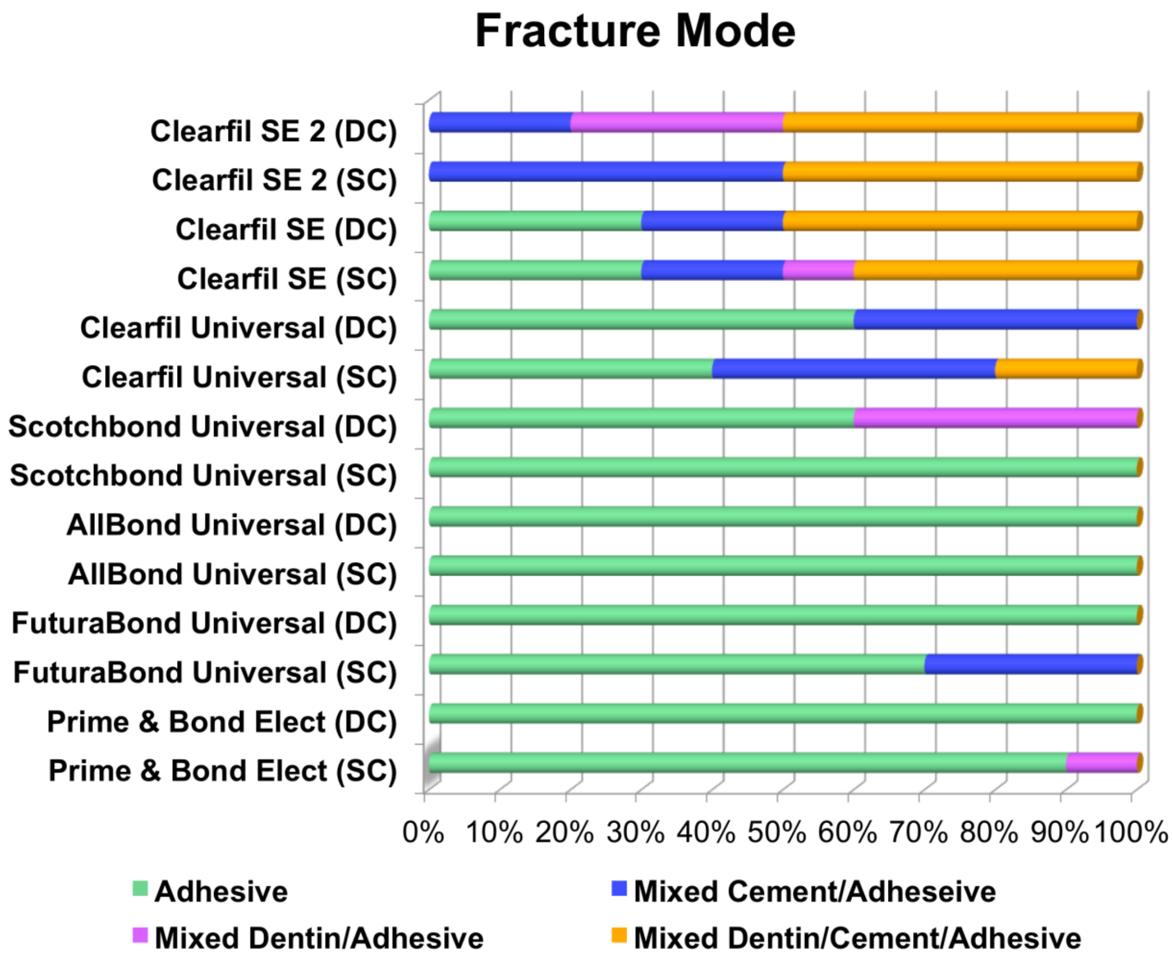
Fracture Mode
Clearfil SE 2 (DC)
Clearfil SE 2 (SC)
Clearfil SE (DC)
Clearfil SE (SC)
Clearfil Universal (DC)
Clearfil Universal (SC)
Scotchbond Universal (DC)
Scotchbond Universal (SC)
AllBond Universal (DC)
AllBond Universal (SC)
FuturaBond Universal (DC)
FuturaBond Universal (SC)
Prime & Bond Elect (DC)
Prime & Bond Elect (SC)
0% 10% 20% 30% 40% 50% 60% 70% 80% 90% 100%
Adhesive
Mixed Cement/Adheseive
Mixed Dentin/Adhesive
Mixed Dentin/Cement/Adhesive

Table 1		
Adhesive	**Components**	**Self-etch strategy with dual or self-cure resins** **(*with dual-cure activator if indicated by the manufacturer's instructions*)**
Clearfil SE Bond	1. **Primer**: HEMA, MDP, hydrophilic aliphatic dimethacrylate, camphorquinone, water, accelerators, dyes, others 2. **Bond**: bis-GMA, HEMA, MDP, hydrophobic aliphatic dimethacrylate, collodial silica, camphorquinone, initiators, accelerators, others	1. Apply primer to tooth surface and leave in place for 20 seconds 2. Evaporate the volatile ingredients with a mild air stream 3. Apply bond to the tooth surface and then create a uniform film using a gentle air stream 4. Light cure for 10 seconds at 1200 mW/cm^2
Clearfil SE Bond 2	1. **Primer**: HEMA, MDP, hydrophilic aliphatic dimethacrylate, camphorquinone, accelerators, water, dyes 2. **Bond**: bis-GMA, HEMA, MDP, hydrophobic aliphatic dimethacrylate, collodial silica, camphorquinone, initiators, accelerators 3. **Dual-cure activator**: ethanol, catalysts, accelerators	1. Apply primer to tooth surface and leave in place for 20 seconds 2. Dry with air stream for >5 seconds until the Primer does not move 3. Dispense 1 drop of each of Bond and Clearfil DC Activator and mix well (use within 90 seconds of mixing) 4. Apply the mixture to the entire cavity wall 5. Dry with air stream for >5 seconds until the Bond does not move 6. Light cure for 10 seconds at 1200 mW/cm^2
All-Bond Universal	1. **Adhesive**: MDP, bis-GMA, HEMA, ethanol, water, initiators	1. Apply two separate coats of adhesive, scrubbing the preparation with a micro brush for 10-15 seconds per coat. Do not light polymerize between coats 2. Evaporate excess solvent by thoroughly air-drying with an air syringe for at least 10 seconds, there should be no visible movement of the material. The surface should have a uniform glossy appearance 3. Light cure for 10 seconds at 1200 mW/cm^2

Table 1		
Adhesive	**Components**	**Self-etch strategy with dual or self-cure resins** (***with dual-cure activator if indicated by the manufacturer's instructions***)
Clearfil Universal	1. **Bond**: bis-GMA, HEMA, ethanol, MDP, hydrophilic aliphatic dimethacrylate, colloidal silica, camphorquinone, silane coupling agent, accelerators, initiators, water 2. **Dual-cure Activator**: ethanol, catalysts, accelerators	1. Dispense 1 drop of Bond and 1 drop of Dual-cure Activator into a mixing well and mix together with applicator brush (must use within 90 seconds of mixing) 2. Apply to cavity surface and leave in place for 5 seconds 3. Dry for >5 seconds until the mixture does not move 4. Light cure for 10 seconds at 1200 mW/cm^2
FuturaBond U	1. **Adhesive**: "Modified" 10-MDP, HEMA, bis-GMA, HEDMA, acidic adhesive monomer, urethane dimethacrylate, catalyst	1. Detach SingleDose blister at the perforation and turn the printed side up 2. Hold the SingleDose blister between thumb and forefinger and, by pressing on the area marked "press here", allow the liquid contained in the blister to flow into the mixing and dispensing chamber 3. Use a Single Tim applicator to pierce into the dispensing chamber and stir thoroughly 4. Apply adhesive to cavity surface and rub in for 20 seconds 5. Dry off for at least 5 seconds to remove solvents 6. Light cure for 10 seconds at 1200 mW/cm^2
Prime&Bond Elect	1. **Adhesive**: acetone, urethane dimethacrylate monomer, dipentaerythritol penta-acrylate phosphate, 2-hydroxy-3-acryloyloxypropyl methacrylate, HEMA 2. **Self-cure Activator**: acetone, urethane dimethacrylate monomer, 2-hydroxyethyl methacrylate, diphenyl (2,4,6-trimethylbenzoyl) phosphine	1. Place 1-2 drops of Adhesive into a well 2. Place equal number of drops of Self-Cure Activator into the same well 3. Mix contacts for 1-2 seconds with a clean, unused brush tip 4. Apply mixture to all tooth surfaces and agitate for 20 seconds 5. Remove excess solvent by air drying for at least 5 seconds 6. Light cure for 10 seconds at 1200 mW/cm^2

Table 1		
Adhesive	**Components**	**Self-etch strategy with dual or self-cure resins (*with dual-cure activator if indicated by the manufacturer's instructions*)**
Scotchbond Universal	1. **Adhesive**: MDP, bis-GMA, HEMA, ethanol, water, initiators 2. **Dual-cure Activator**: ethyl alcohol, sodium p-toluenesulfinate, methyl ethyl ketone	1. Place 1 drop of each Adhesive and Dual-cure Activator into a mixing well and mix for 5 seconds 2. Apply the mixture to the cavity surface and rub it in for 20 seconds 3. Apply air stream for about 5 seconds until mixture no longer moves 4. Light cure for 10 seconds at 1200 mW/cm^2

Bonding Agent	Mean Shear Bond Strength MPa (st dev)		
	Dual Cure	**Self Cure**	**Total Mean**
Clearfil SE Bond 2	27.5 (6.0)	20.2 (5.3)	**23.8 (6.7) a**
Clearfil SE Bond	19.2 (5.2)	18.1 (4.0)	**18.6 (4.5) b**
Clearfil Universal	13.1 (3.8)	9.7 (6.9)	**11.4 (5.7) c**
Scotchbond Universal	8.1 (5.7)	7.6 (5.0)	**7.9 (5.2) c,d**
All-Bond Universal	6.7 (4.7)	5.2 (4.7)	**5.9 (4.6) d,e**
FuturaBond U	5.4 (5.3)	4.2 (5.0)	**4.7 (5.1) d,e**
Prime&Bond Elect	1.3 (1.0)	3.2 (3.5)	**2.2 (2.7) e**
Total Mean	**11.6 (9.6) A**	**9.7 (7.9) B**	
Groups with the same lower case letter per column or upper case letter per row are not significantly different (P>0.05)			

Table 2